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(54) Title: **GREEN LIGHT EMITTING ELECTROLUMINESCENT MATERIAL**

(57) Abstract: An electroluminescent device which emits substantially saturated green light has a layer of Tris (2, 2, 6, 6- tetramethyl-3,5-heptanedionato) Terbium (III) OPNP and a layer of BCP.

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Green Light Emitting Electroluminescent Material

The present invention relates to electroluminescent devices.

5 Materials which emit light when an electric current is passed through them are well known and used in a wide range of display applications. Liquid crystal devices and devices which are based on inorganic semiconductor systems are widely used, however these suffer from the disadvantages of high energy consumption, high cost of manufacture, low quantum efficiency and the inability to make flat panel displays.

10

Organic polymers have been proposed as useful in electroluminescent devices, but it is not possible to obtain pure colours, they are expensive to make and have a relatively low efficiency.

15 Another compound which has been proposed is aluminium quinolate, but this requires dopants to be used to obtain a range of colours and has a relatively low efficiency.

In an article in Chemistry letters pp 657-660, 1990 Kido et al disclosed that a terbium
20 III acetyl acetonate complex was green electroluminescent and in an article in Applied Physics letters 65 (17) 24 October 1994 Kido et al disclosed that a europium III triphenylene diamine complexes was red electroluminescent but these were unstable in atmospheric conditions and difficult to produce as films.

25 Patent application WO98/58037 describes a range of lanthanide complexes which can be used in electroluminescent devices which have improved properties and give better results. Patent Applications PCT/GB98/01773, PCT/GB99/03619, PCT/GB99/04030, PCT/GB99/04024, PCT/GB99/04028, PCT/GB00/00268 describe electroluminescent complexes, structures and devices using rare earth chelates.

30

US Patent 5128587 discloses an electroluminescent device which consists of an organometallic complex of rare earth elements of the lanthanide series sandwiched between a transparent electrode of high work function and a second electrode of low work function with a hole conducting layer interposed between the electroluminescent layer and the transparent high work function electrode and an electron conducting layer interposed between the electroluminescent layer and the electron injecting low work function anode.

The characteristics of the emitted light such as its wavelength distribution, frequency, phase, intensity etc. and the properties of the device such as its efficiency, power consumption, optimum voltage brightness, speed of response temperature stability etc. depend on the selection from a wide range of variables such as the selection of the metal, the selection of the ligands, the nature of the electrodes and any layers formed on the electrodes, the hole transmitting and the electron donating or transmitting materials etc. In order to obtain an electroluminescent device or structure with a specific property or properties particular compounds or materials need to be selected from a wide range of variables and it has been found that the selection of a material or materials to enhance one property can have a deleterious effect on other properties.

20

We have now devised an electroluminescent material which emits a high intensity green light of high purity.

Green light of high purity is needed for devices in which a colour picture is formed from the combination of light of each of the three primary colours and this can give a complete range of colours. The nearer each of the primary colours is to the pure or saturated colour the easier it is to obtain pictures with "true" colours.

According to the invention there is provided an electroluminescent device which comprises (i) a layer of a Tb(III)chelate and (ii) a layer of BCP, which structure emits a substantially saturated green light when an electric current is passed through it.

- 5 By a substantially saturated green light is meant light which falls on or near the boundary line in the green spectrum on a colour chromacity chart. A chart is shown in fig. 18 and saturated green is on or close to the boundary line at 550nm. In the present invention by substantially saturated green light is meant light which has is within the boundary line (1) in fig. 18 of the accompanying drawings.

10

The invention also provides an electroluminescent device which comprises (i) a first electrode, (ii) a layer of a hole transmitting material, (iii) an electroluminescent layer comprising a Tb(III)chelate (iv) a BCP layer, (v) a layer of an electron transmitting material and (vi) a second electrode.

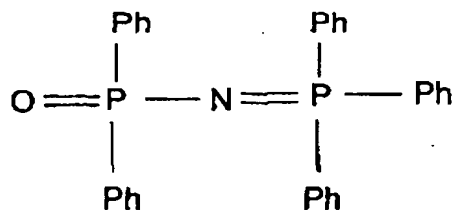
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BCP is bathocupron.

The terbium(III) chelate preferably is of formula $Tb(L_a)_3(L_p)$ where (L_a) is preferably tmhd [tris (2,2,6,6- tetramethyl-3,5- heptanedionato)] and (L_p) is OPNP where OPNP

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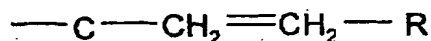
includes OPNP itself and



(I)

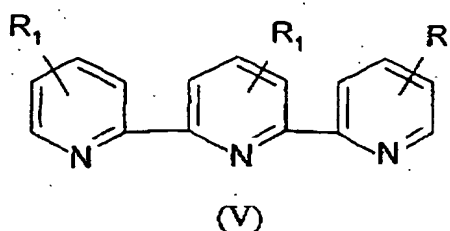
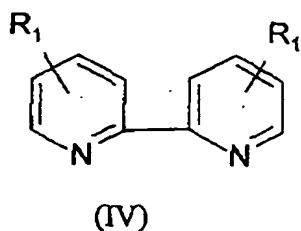
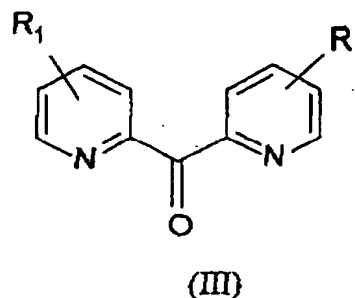
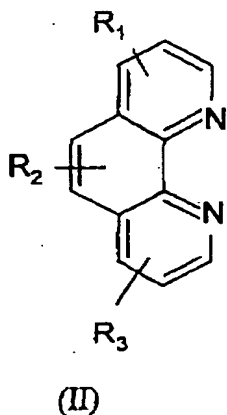
- 25 where each Ph which can be the same or different and can be a phenyl (OPNP) or a substituted phenyl group, other substituted or unsubstituted aromatic group, a

- substituted or unsubstituted heterocyclic or polycyclic group, a substituted or unsubstituted fused aromatic group such as a naphthyl, anthracene, phenanthrene, perylene or pyrene group. The substituents can be for example an alkyl, aralkyl, alkoxy, aromatic, heterocyclic, polycyclic group, halogen such as fluorine, cyano, amino and substituted amino groups etc. Examples are given in figs. 1 to 3 of the drawings where R, R₁, R₂, R₃ and R₄ can be the same or different and are selected from hydrogen, hydrocarbyl groups, substituted and unsubstituted aromatic, heterocyclic and polycyclic ring structures, fluorocarbons such as trifluoromethyl groups, halogens such as fluorine or thiophenyl groups; R, R₁, R₂, R₃ and R₄ can also form substituted and unsubstituted fused aromatic, heterocyclic and polycyclic ring structures and can be copolymerisable with a monomer e.g. styrene. R, R₁, R₂, R₃ and R₄ can also be unsaturated alkylene groups such as vinyl groups or groups



where R is as above.

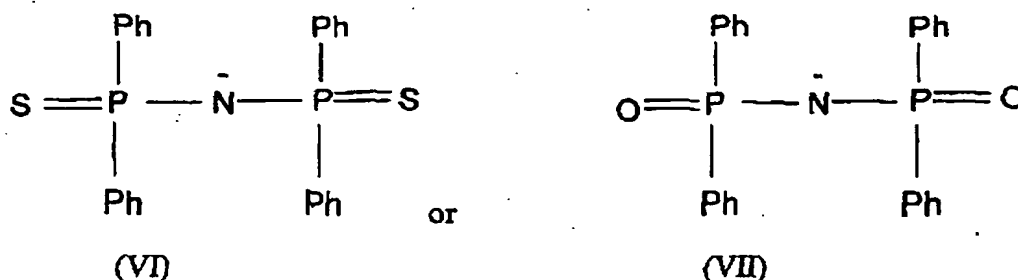
- 15 L_p can also be compounds of formulae



where R_1 , R_2 and R_3 are as referred to above, for example bathophen shown in fig. 3 of the drawings in which R is as above.

L_p can also be

5



where Ph is as above.

10

Other examples of L_p are as shown in figs. 4 to 8

Preferably the Tb(III)chelate comprises Tris (2,2,6,6- tetramethyl-3,5-heptanedionato) Terbium (III) OPNP, $[\text{Tb}(\text{tmhd})_3\text{OPNP}]$ wher OPNP is (I) above.

15

The first electrode is preferably a transparent substrate which is a conductive glass or plastic material which acts as the anode, preferred substrates are conductive glasses such as indium tin oxide coated glass, but any glass which is conductive or has a conductive layer can be used. Conductive polymers and conductive polymer coated glass or plastics materials can also be used as the substrate.

20

Optionally there is a layer of an electron transmitting material between the cathode and the electroluminescent material layer, the electron transmitting material is a material which will transport electrons when an electric current is passed through electron transmitting materials include a metal complex such as a metal quinolate e.g. an aluminium quinolate, lithium quinolate a cyano anthracene such as 9,10 dicyano

25

anthracene, a polystyrene sulphonate and compounds of formulae shown in figs. 9 and 10. Instead of being a separate layer the electron transmitting material can be mixed with the electroluminescent material and co-deposited with it.

- 5 In general the thickness of the layers is from 5nm to 500nm and preferably the thickness of the electroluminescent layer is from 20 to 50nm.

- The second electrode functions as the cathode and can be any low work function metal e.g. aluminium, calcium, lithium, silver/magnesium alloys etc., aluminium is a
10 preferred metal. Lithium fluoride can be used as the second electrode for example by having a lithium fluoride layer formed on a metal.

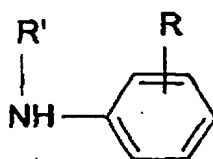
- Preferably there is a hole transporting layer deposited on the transparent substrate and the electroluminescent material is deposited on the hole transporting layer. The hole
15 transporting layer serves to transport holes and to block the electrons, thus preventing electrons from moving into the electrode without recombining with holes. The recombination of carriers therefore mainly takes place in the emitter layer.

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20 the electroluminescent material is deposited on the hole transporting layer. The hole transporting layer serves to transport holes and to block the electrons, thus preventing electrons from moving into the electrode without recombining with holes. The recombination of carriers therefore mainly takes place in the emitter layer.

- 25 Hole transporting layers are used in polymer electroluminescent devices and any of the known hole transporting materials in film form can be used.

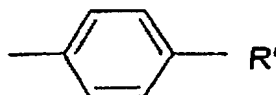
- Examples of such hole transporting materials are aromatic amine complexes such as poly (vinylcarbazole), N, N'-diphenyl-N, N'-bis (3-methylphenyl) -1,1' -biphenyl -
30 4,4'-diamine (TPD), an unsubstituted or substituted polymer of an amino substituted

aromatic compound, a polyaniline, substituted polyanilines, polythiophenes, substituted polythiophenes, polysilanes etc. Examples of polyanilines are polymers of



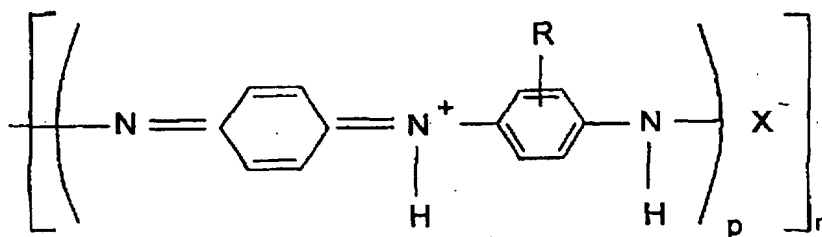
(VIII)

where R is in the ortho - or meta-position and is hydrogen, C1-18 alkyl, C1-6 alkoxy, amino, chloro, bromo, hydroxy or the group



where R is alkyl or aryl and R' is hydrogen, C1-6 alkyl or aryl with at least one other monomer of formula I above.

15 Polyanilines which can be used in the present invention have the general formula



(XIX)

where p is from 1 to 10 and n is from 1 to 20, R is as defined above and X is an anion, preferably selected from Cl, Br, SO₄, BF₄, PF₆, H₂PO₃, H₂PO₄, arylsulphonate, arenedicarboxylate, polystyrenesulphonate, polyacrylate

alkylsulphonate, vinylsulphonate, vinylbenzene sulphonate, cellulose sulphonate, camphor sulphonates, cellulose sulphate or a perfluorinated polyanion.

5 Examples of arylsulphonates are p-toluenesulphonate, benzenesulphonate, 9,10-anthraquinone-sulphonate and anthracenesulphonate, an example of an arenedicarboxylate is phthalate and an example of arenecarboxylate is benzoate.

10 We have found that protonated polymers of the unsubstituted or substituted polymer of an amino substituted aromatic compound such as a polyaniline are difficult to evaporate or cannot be evaporated, however we have surprisingly found that if the unsubstituted or substituted polymer of an amino substituted aromatic compound is deprotonated the it can be easily evaporated i.e. the polymer is evaporable.

15 Preferably evaporable deprotonated polymers of unsubstituted or substituted polymer of an amino substituted aromatic compound are used. The de-protonated unsubstituted or substituted polymer of an amino substituted aromatic compound can be formed by deprotonating the polymer by treatment with an alkali such as ammonium hydroxide or an alkali metal hydroxide such as sodium hydroxide or potassium hydroxide.

20

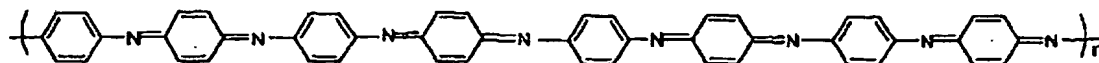
The degree of protonation can be controlled by forming a protonated polyaniline and de-protonating. Methods of preparing polyanilines are described in the article by A. G. MacDiarmid and A. F. Epstein, Faraday Discussions, Chem Soc.88 P319 1989.

25 The conductivity of the polyaniline is dependant on the degree of protonation with the maximum conductivity being when the degree of protonation is between 40 and 60% e.g. about 50% for example.

Preferably the polymer is substantially fully deprotonated

30

A polyaniline can be formed of octamer units i.e. p is four e.g.



5 The polyanilines can have conductivities of the order of 1×10^{-1} Siemen cm^{-1} or higher.

The aromatic rings can be unsubstituted or substituted e.g. by a C1 to 20 alkyl group such as ethyl.

10 The polyaniline can be a copolymer of aniline and preferred copolymers are the copolymers of aniline with o-anisidine, m-sulphanilic acid or o-aminophenol, or o-toluidine with o-aminophenol, o-ethylaniline, o-phenylene diamine or with amino anthracenes.

15 Other polymers of an amino substituted aromatic compound which can be used include substituted or unsubstituted polyaminonaphthalenes, polyaminoanthracenes, polyaminophenanthrenes, etc. and polymers of any other condensed polyaromatic compound. Polyaminoanthracenes and methods of making them are disclosed in US Patent 6,153,726. The aromatic rings can be unsubstituted or substituted e.g. by a
20 group R as defined above.

The polyanilines can be deposited on the first electrode by conventional methods e.g. by vacuum evaporation, spin coating, chemical deposition, direct electrodeposition etc. preferably the thickness of the polyaniline layer is such that the layer is
25 conductive and transparent and can is preferably from 20nm to 200nm. The polyanilines can be doped or undoped, when they are doped they can be dissolved in a solvent and deposited as a film, when they are undoped they are solids and can be deposited by vacuum evaporation i.e. by sublimation.

The polymers of an amino substituted aromatic compound such as polyanilines referred to above can also be used as buffer layers with or in conjunction with other hole transporting materials.

- 5 The structural formulae of some other hole transporting materials are shown in Figures 11 to 16 of the drawings, where R_1 , R_2 and R_3 can be the same or different and are selected from hydrogen, and substituted and unsubstituted hydrocarbyl groups such as substituted and unsubstituted aliphatic groups, substituted and unsubstituted aromatic, heterocyclic and polycyclic ring structures, fluorocarbons such as trifluoryl methyl groups, halogens such as fluorine or thiophenyl groups; R_1 , R_2 and R_3 can also form substituted and unsubstituted fused aromatic, heterocyclic and polycyclic ring structures and can be copolymerisable with a monomer e.g. styrene. X is Se, S or O, Y can be hydrogen, substituted or unsubstituted hydrocarbyl groups, such as substituted and unsubstituted aromatic, heterocyclic and polycyclic ring structures, fluorine, fluorocarbons such as trifluoryl methyl groups, halogens such as fluorine or thiophenyl groups or nitrile.

- Examples of R_1 and/or R_2 and/or R_3 include aliphatic, aromatic and heterocyclic alkoxy, aryloxy and carboxy groups, substituted and substituted phenyl, fluorophenyl, biphenyl, phenanthrene, anthracene, naphthyl and fluorene groups alkyl groups such as t-butyl, heterocyclic groups such as carbazole.

The electroluminescent material produces a saturated green light when an electric current is passed through it, the voltage applied is preferably up to 12 volts

25

The preferred thicknesses of the layers are :-

hole transmitting material layer $40\text{nm} \pm 2$

Tb(III)chelate layer $50\text{nm} \pm 3$

BCP layer $6\text{nm} \pm 2$

- 30 Alq₃ layer $20\text{nm} \pm 5$

The invention is illustrated in the Example.

Example

5

An ITO coated glass piece ($1 \times 1 \text{ cm}^2$) had a portion etched out with concentrated hydrochloric acid to remove the ITO and was cleaned and dried. The device was fabricated by sequentially forming on the ITO, by vacuum evaporation, a device as shown in fig. 17 in which (1) is ITO, (2) is CuPc (3) is NPB (4) is Tb(tmhd)₃OPNP (5) is BCP (6) is Alq₃ (7) is LiF and (8) is Al layers comprising:-

10

ITO | CuPc (2nm) | NPB(40nm) | Tb(tmhd)₃OPNP (50nm) | BCP (6nm) | Alq₃(20nm) | LiF(0.7nm) | Al (150nm).

15 Where CuPc is copper phthalocyanine, NPB is as shown in fig. 5, OPNP is as in (I), BCP is bathocupron and Alq₃ is aluminium quinolate.

The organic coating on the portion which had been etched with the concentrated hydrochloric acid was wiped with a cotton bud.

20

The coated electrodes were stored in a vacuum desiccator over a molecular sieve and phosphorous pentoxide until they were loaded into a vacuum coater (Edwards, 10^{-6} torr) and aluminium top contacts made. The active area of the LED's was 0.08 cm by 0.1 cm^2 the devices were then kept in a vacuum desiccator until the electroluminescence studies were performed.

25

The ITO electrode was always connected to the positive terminal. The current vs. voltage studies were carried out on a computer controlled Keithly 2400 source meter.

30

- 12 -

An electric current of 12 volts was passed through the structure and the light emitted is shown on the chart of fig. 18 which is saturated green light, the colour of the light emitted by typical terbium chelates and by aluminium quinolate is shown for comparison

5

Claims

1. An electroluminescent device which comprises (i) a layer of a Tb(III)chelate and (ii) a layer of BCP, which structure emits a saturated green light when an electric current is passed through it.
2. An electroluminescent device as claimed in claim 1 in which the Tb(III) chelate is Tb(tmhd)₃OPNP where tmhd and OPNP are as herein defined.
3. An electroluminescent device as claimed in claim 1 or 2 which comprises (i) a first electrode, (ii) a layer of a hole transmitting material, (iii) an electroluminescent layer comprising a Tb(III)chelate (iv) a BCP layer, (v) a layer of an electron transmitting material and (vi) a second electrode.
4. An electroluminescent device as claimed in claim 3 in which the hole transmitting layer is an aromatic amine complex.
5. An electroluminescent device as claimed in claim 4 in which the hole transmitting layer is formed from a poly(vinylcarbazole), N,N'-diphenyl-N,N'-bis (3-methylphenyl) -1,1' -biphenyl -4,4'-diamine (TPD), polyaniline, or a substituted polyaniline.
6. An electroluminescent device as claimed in claim 5 in which the hole transmitting material has the formula of any of figures 11 to 16.
7. An electroluminescent device as claimed in any one of claims 3 to 6 in which the electron transmitting material is a metal quinolate, a cyano anthracene, 9,10 dicyano anthracene, a polystyrene sulphonate or a compound of formulae as shown in fig. 9 or 10.

8. An electroluminescent device as claimed in claim 7 in which the metal quinolate is lithium, sodium, potassium, zinc, magnesium or aluminium quinolate.
9. An electroluminescent device as claimed in any one of claims 1 to 8 in which the
5 thickness of the Tb(III)chelate layer is from 20 to 50nm.
10. An electroluminescent device as claimed in any one of claims 1 to 9 in which there is a buffer layer between the first electrode and the layer of a hole transmitting material.
- 10 11. An electroluminescent device as claimed in any one of claims 3 to 10 in which the thickness of the layers are:- hole transmitting material layer $40\text{nm} \pm 2$, Tb(III)chelate layer $50\text{nm} \pm 3$, BCP layer $6\text{nm} \pm 2$ and electron transmitting layer $20\text{nm} \pm 5$.
- 15 12. An electroluminescent device as claimed in any one of claims 3 to 11 in which there is a copper phthalocyanine layer on the first layer and a lithium fluoride layer on the second electrode.

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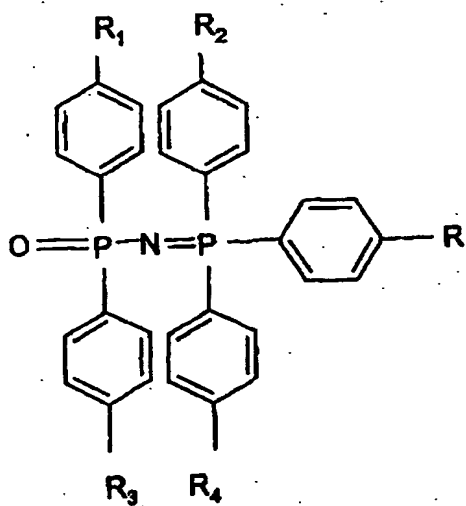


Fig. 1

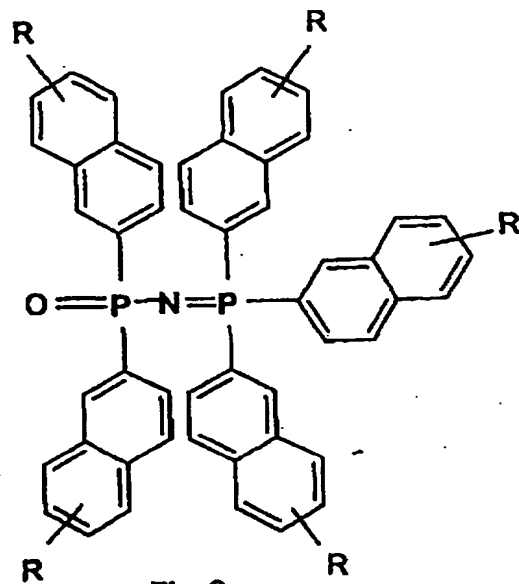


Fig. 2a

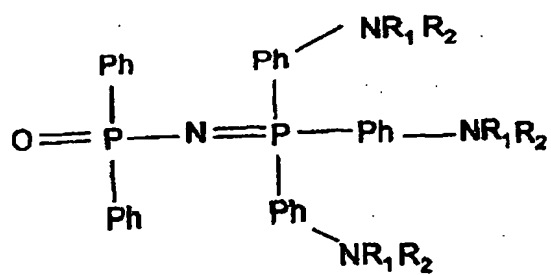


Fig. 2b

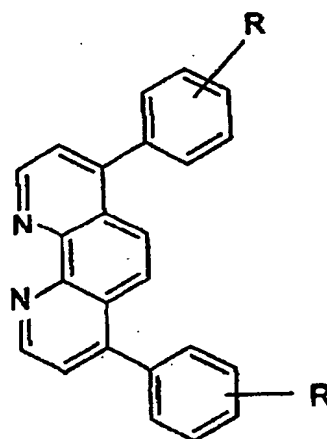


Fig. 3

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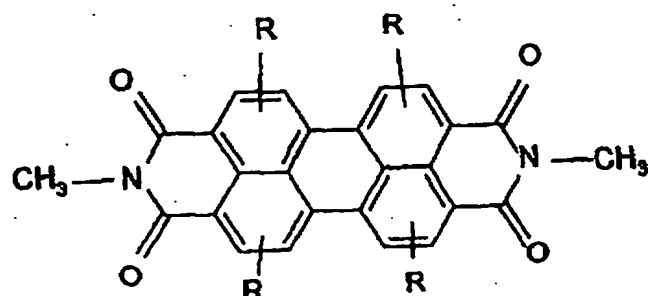


Fig. 4a

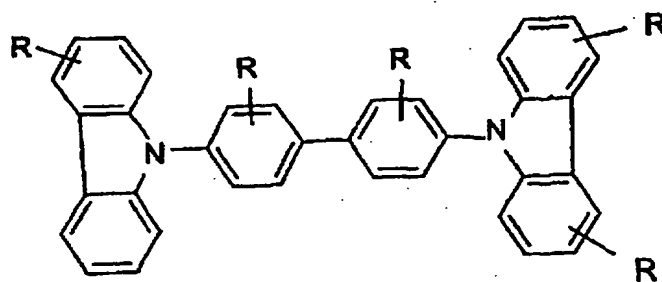


Fig. 4b

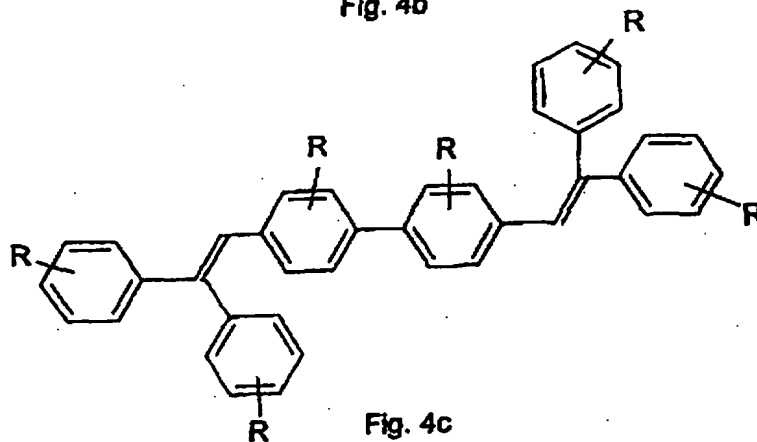


Fig. 4c

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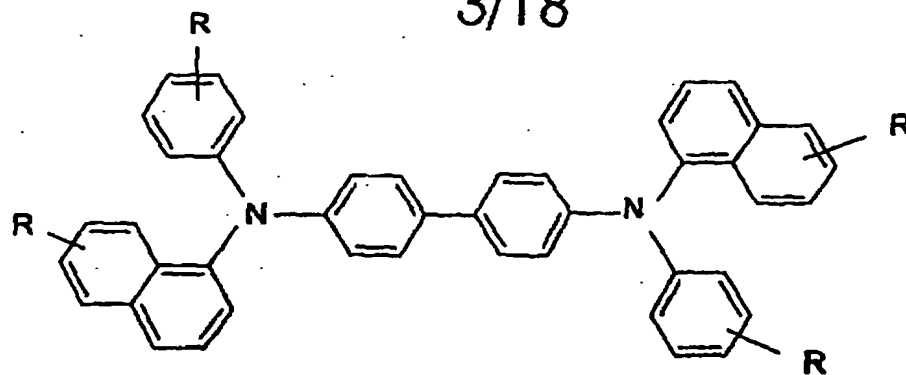


Fig. 4d

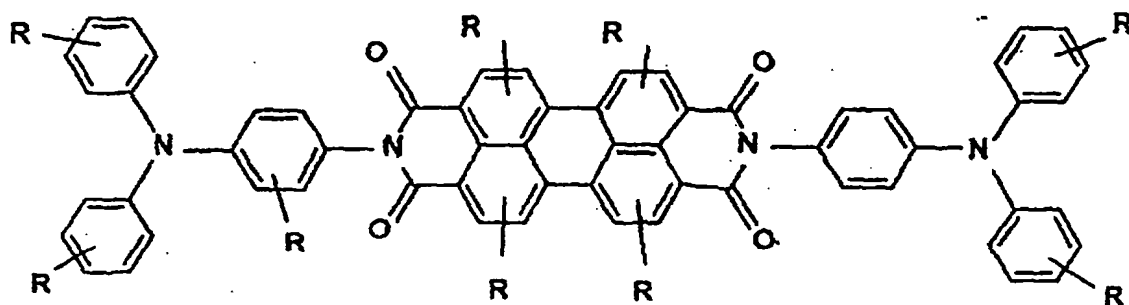


Fig. 4e

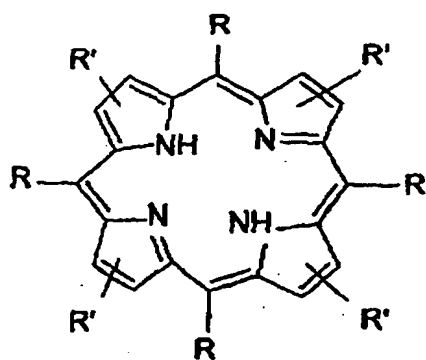


Fig. 4f

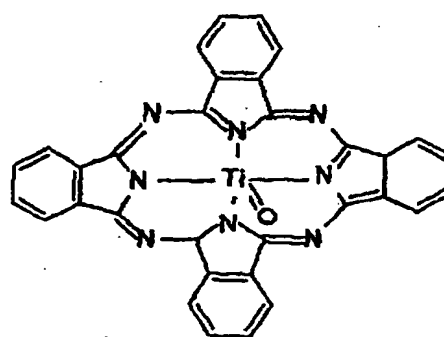


Fig. 4g

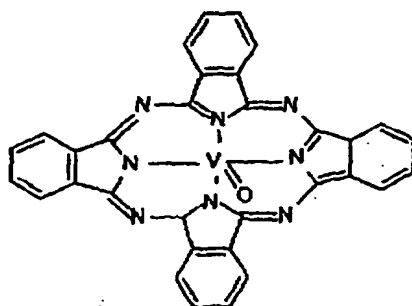


Fig. 4h

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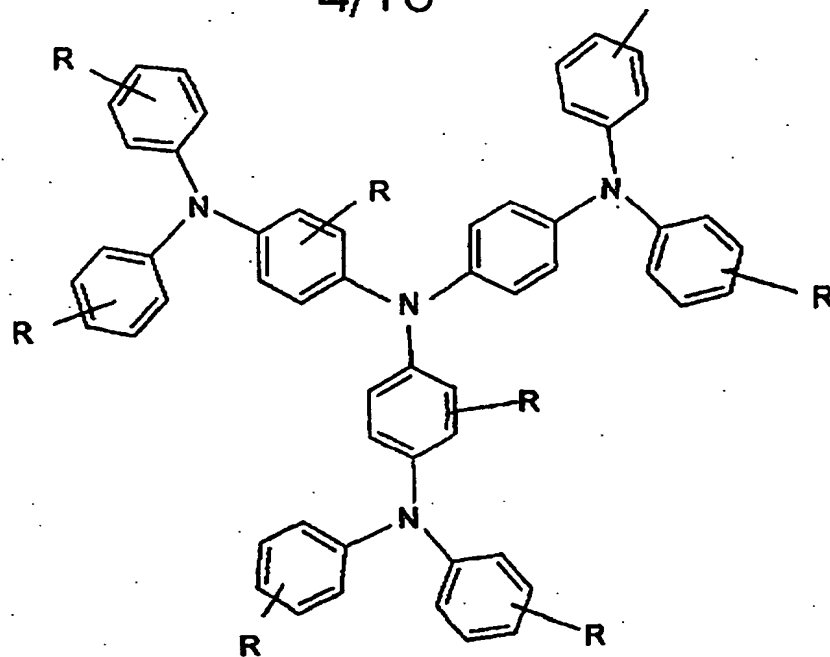


Fig. 4i

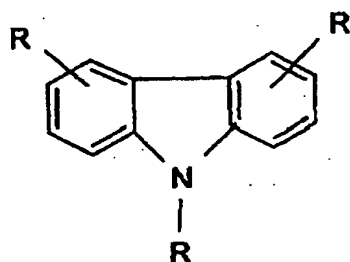


Fig. 4j

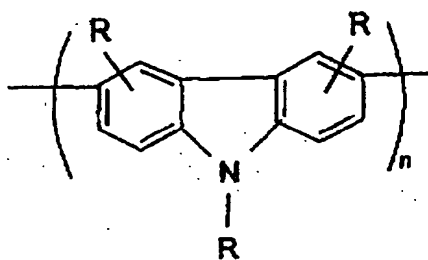


Fig. 4k

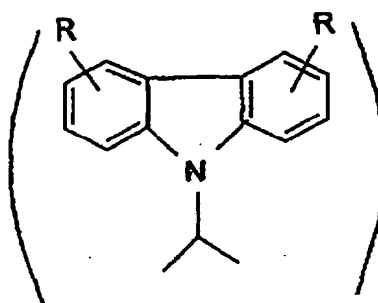


Fig. 4l

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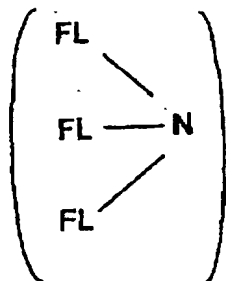


Fig. 5a

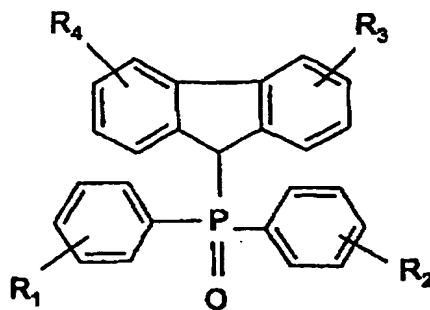


Fig. 5b

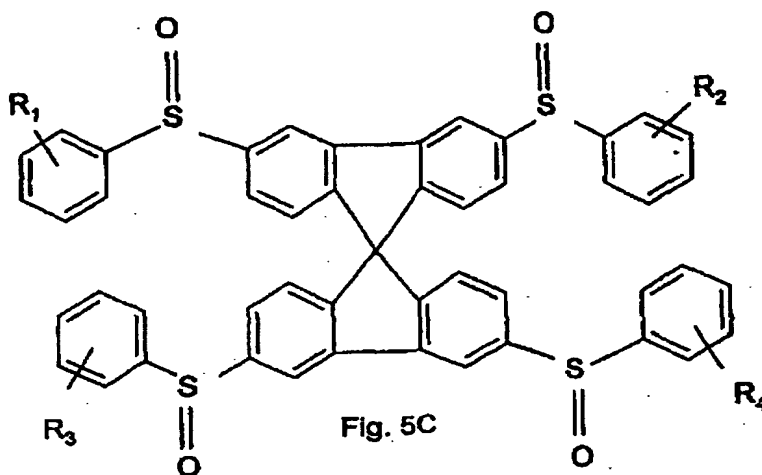


Fig. 5c

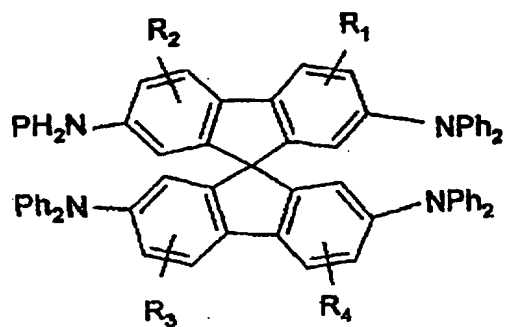


Fig. 5d

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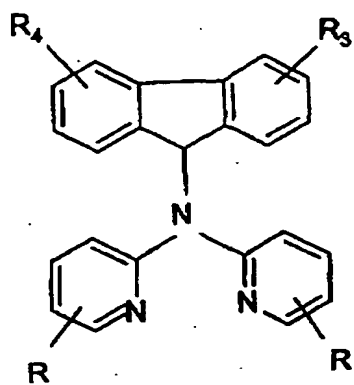


Fig. 5f

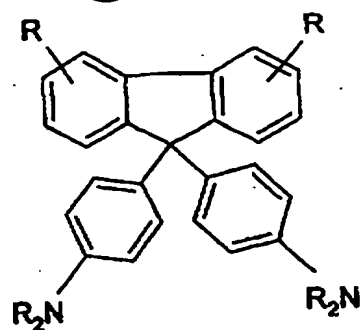


Fig. 5g

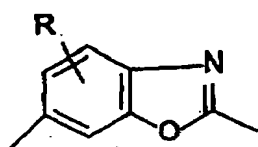


Fig. 6a

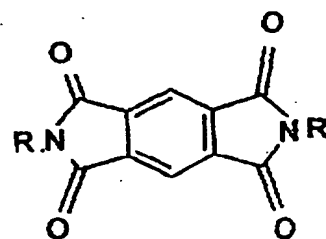


Fig. 6b

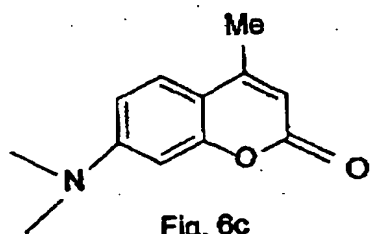


Fig. 6c

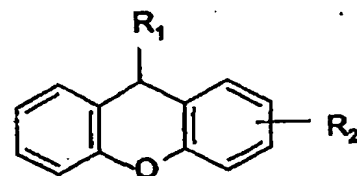


Fig. 6d

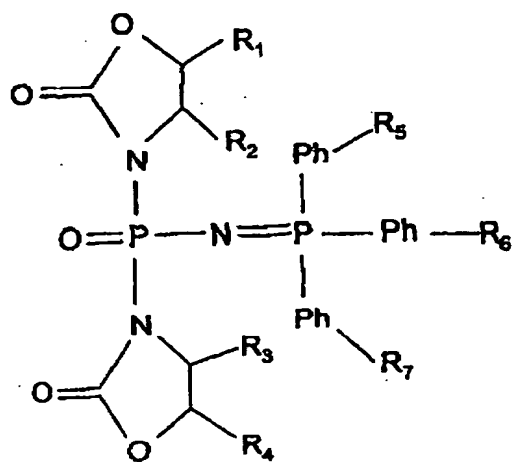


Fig. 6e

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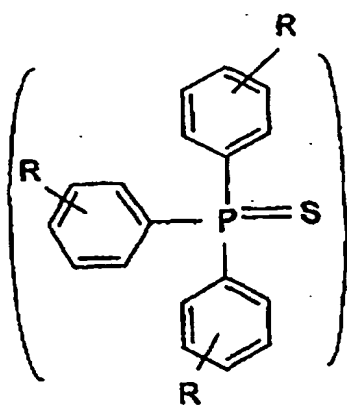


Fig. 7a

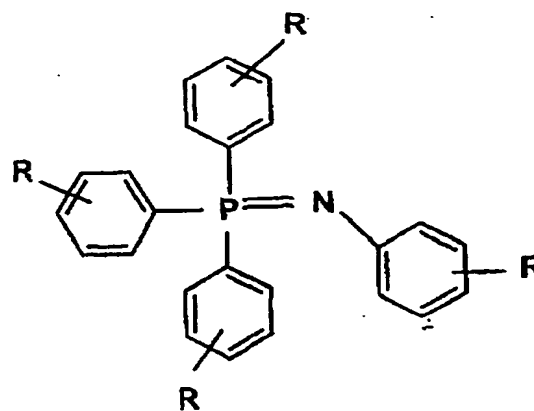


Fig. 7b

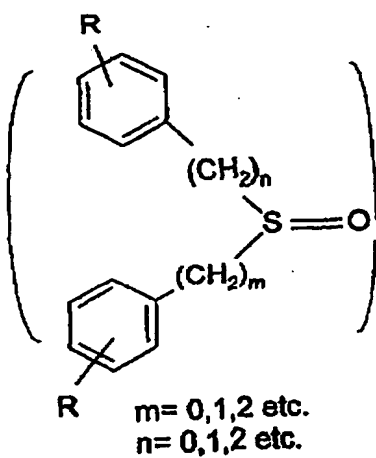


Fig. 7c

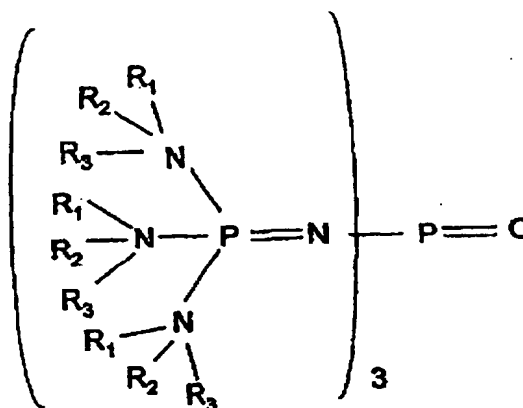


Fig. 7d

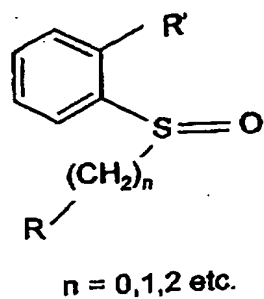


Fig. 7e

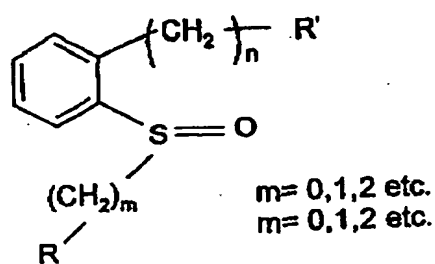


Fig. 7f

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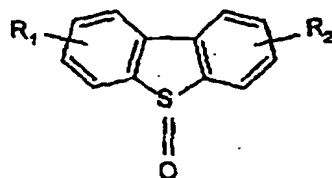


Fig. 8a

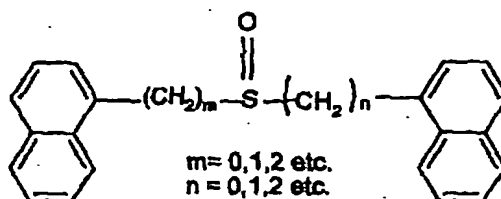


Fig. 8b

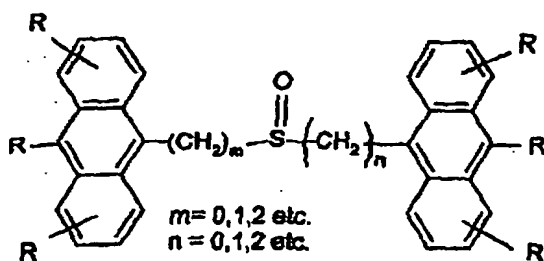


Fig. 8c

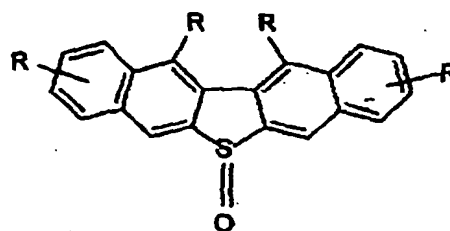


Fig. 8d

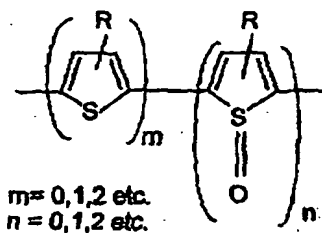


Fig. 8e

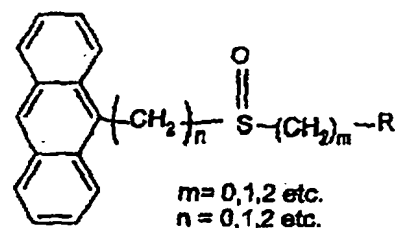


Fig. 8f

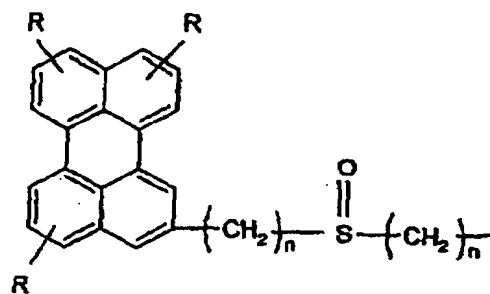


Fig. 8g

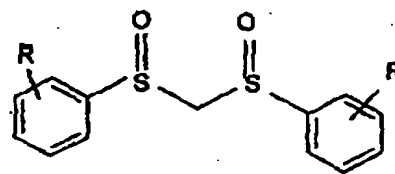
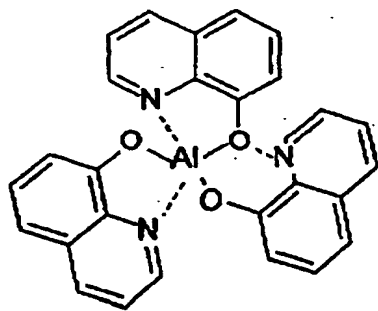
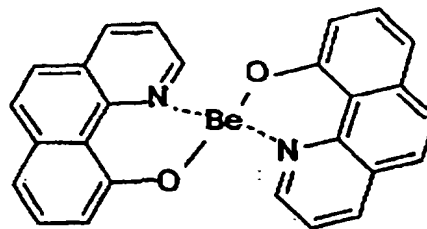


Fig. 8h

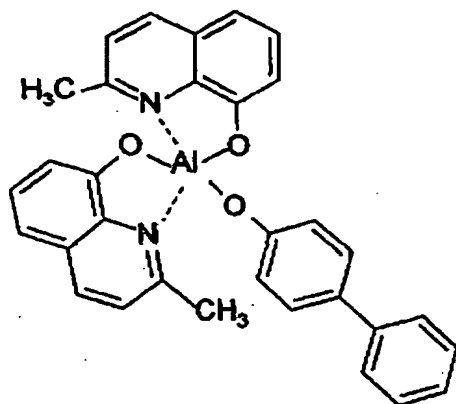
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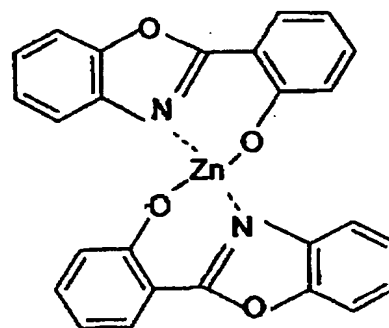
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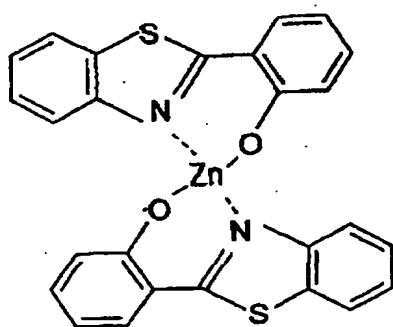
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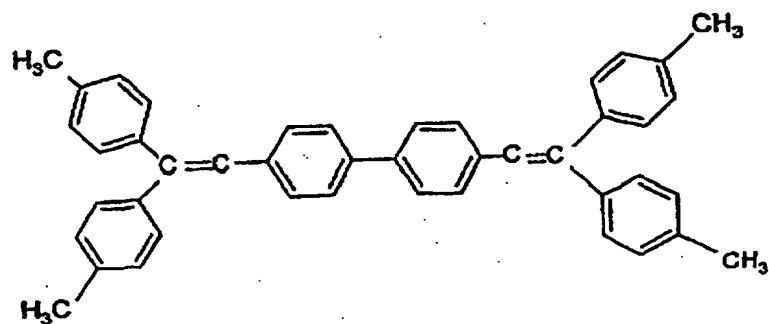
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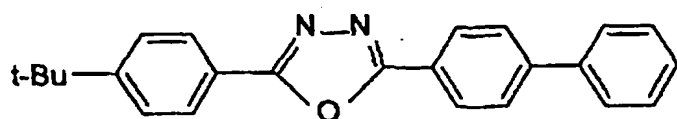
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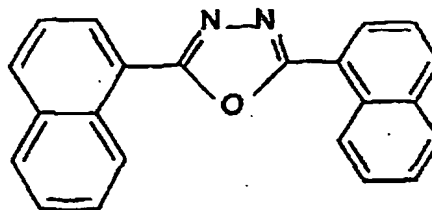
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Fig. 9

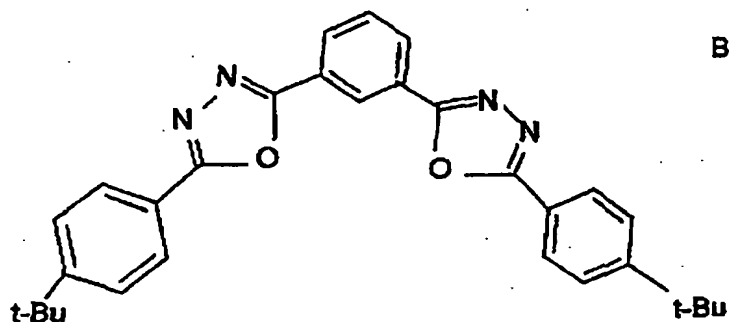
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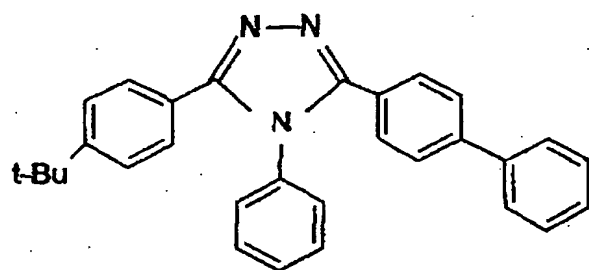
t-Bu-PBD



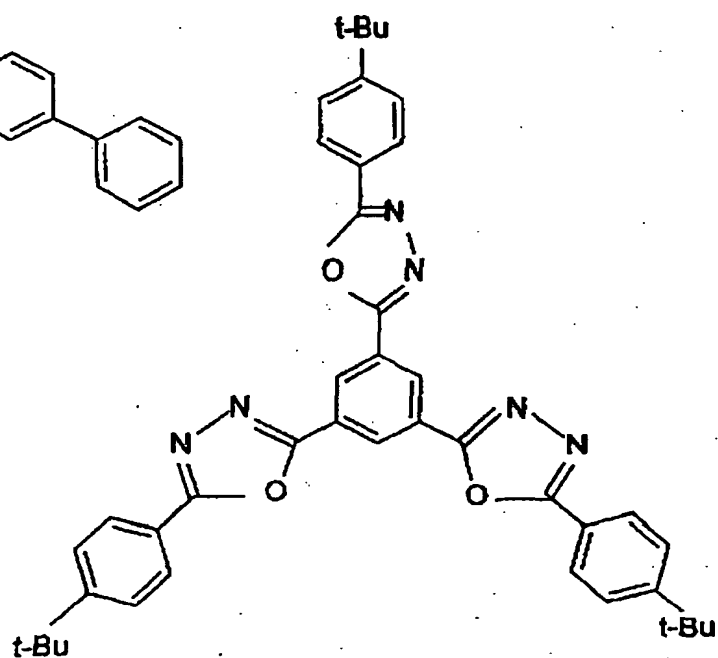
BND



OXD-7



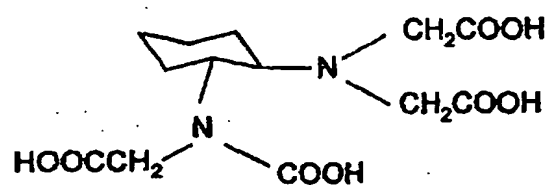
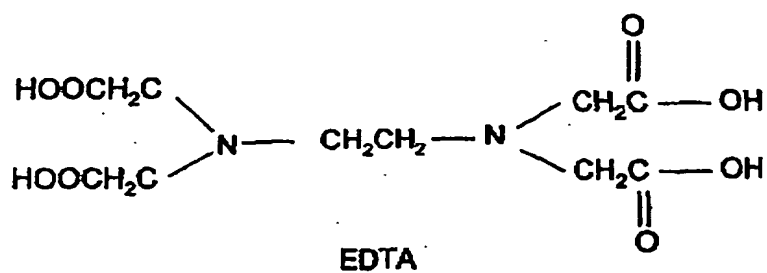
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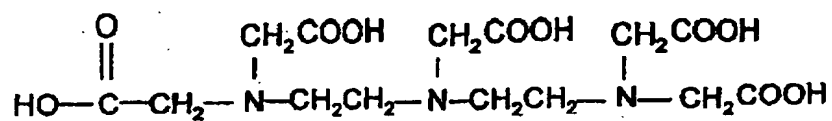
OXD-Star

Fig. 10

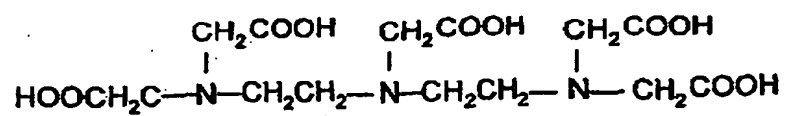
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DCTA



DTPA



TTHA

Fig. 11

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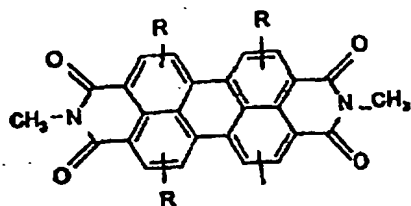


Fig. 12a

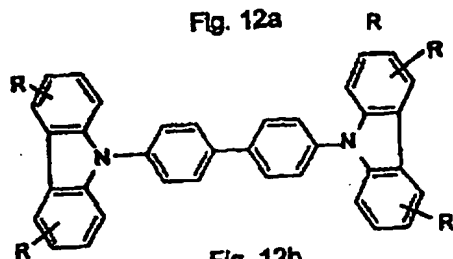


Fig. 12b

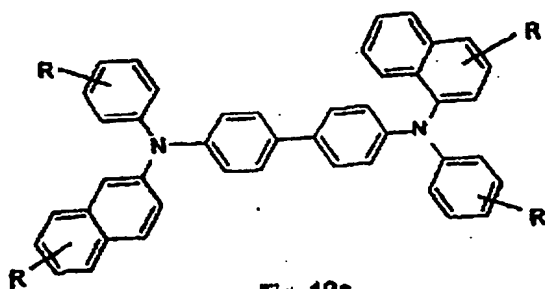


Fig. 12c

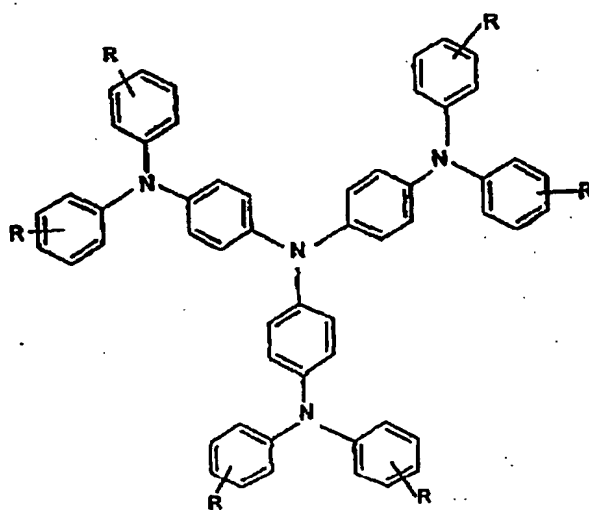


Fig. 12d

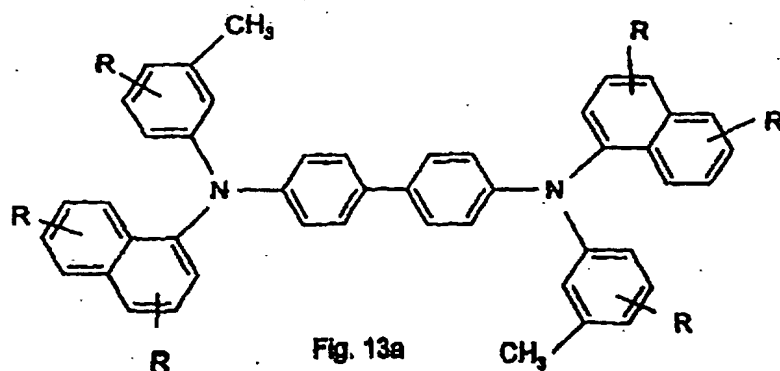


Fig. 13a

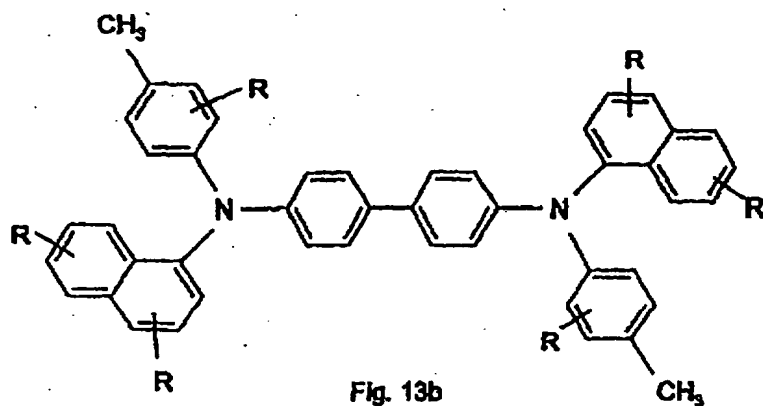


Fig. 13b

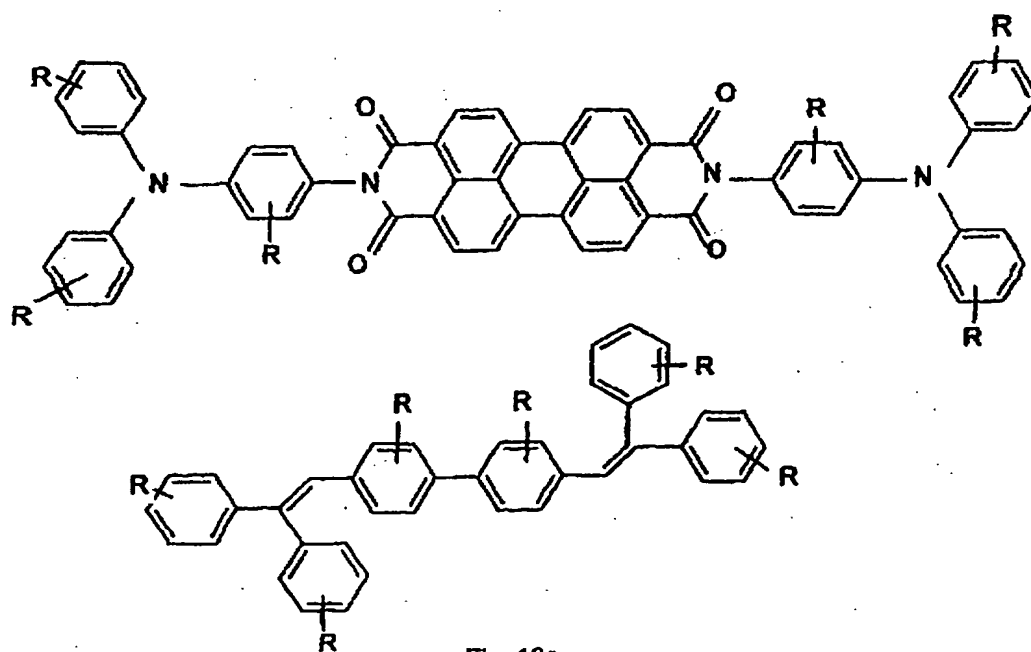


Fig. 13c

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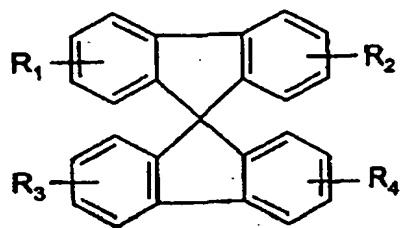


Fig. 14a

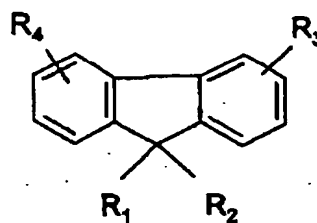
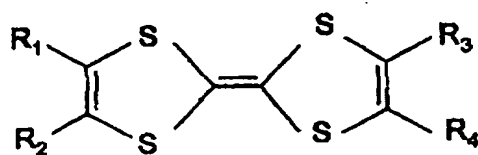


Fig. 14b



or

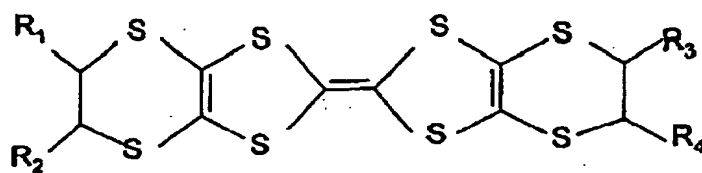


Fig. 14c

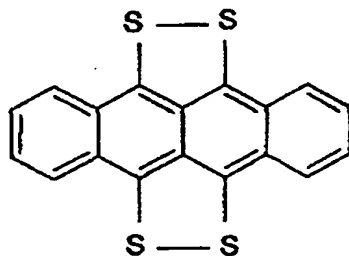


Fig. 14d

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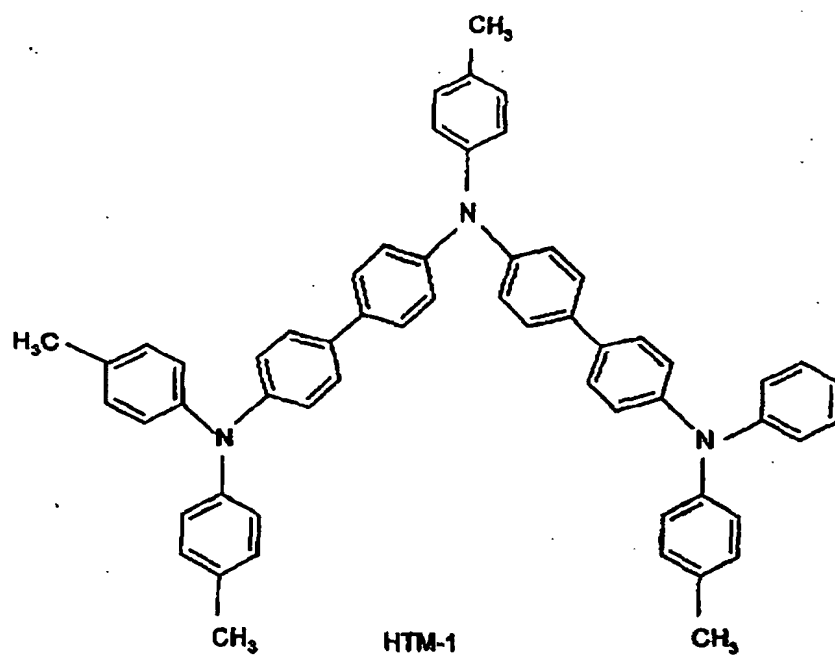


Fig. 15a

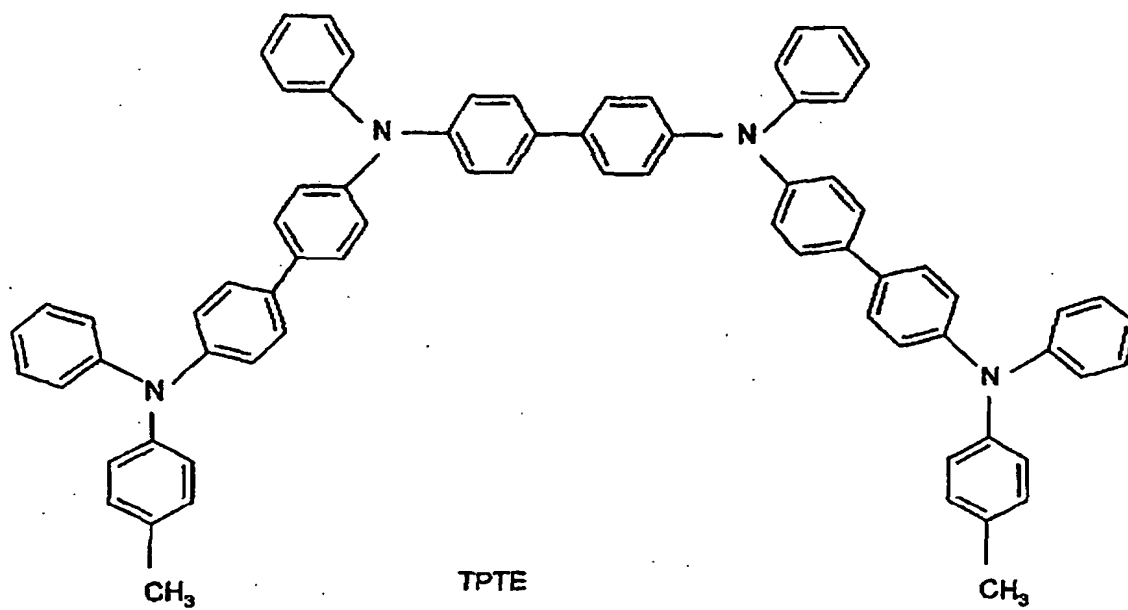


Fig. 15b

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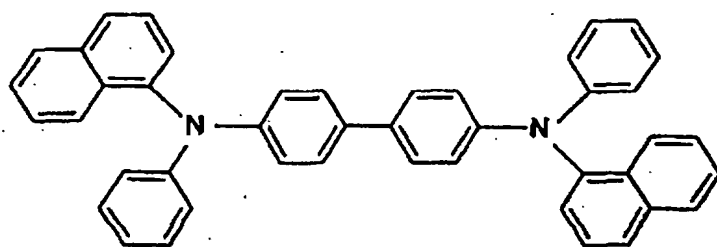
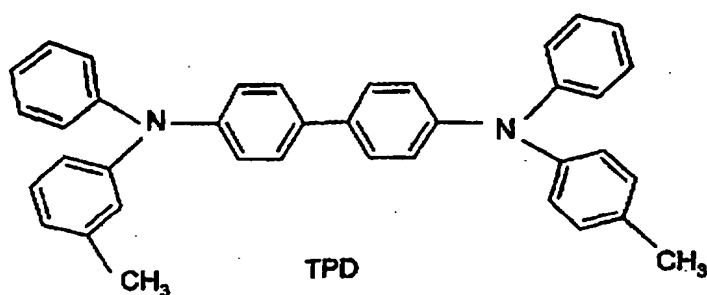
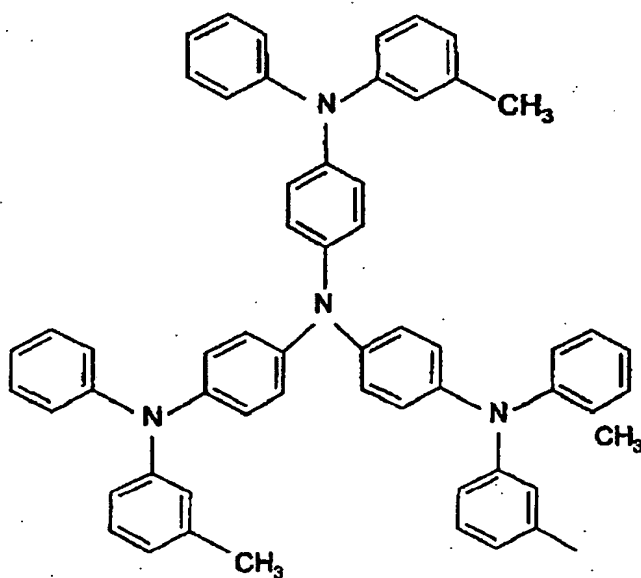
 α -NPB

Fig. 16a



TPD

Fig. 16b



mTADATA

Fig. 16c

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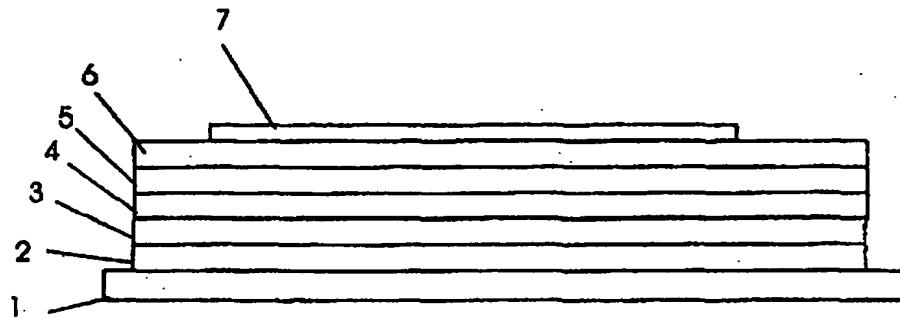


Fig. 17

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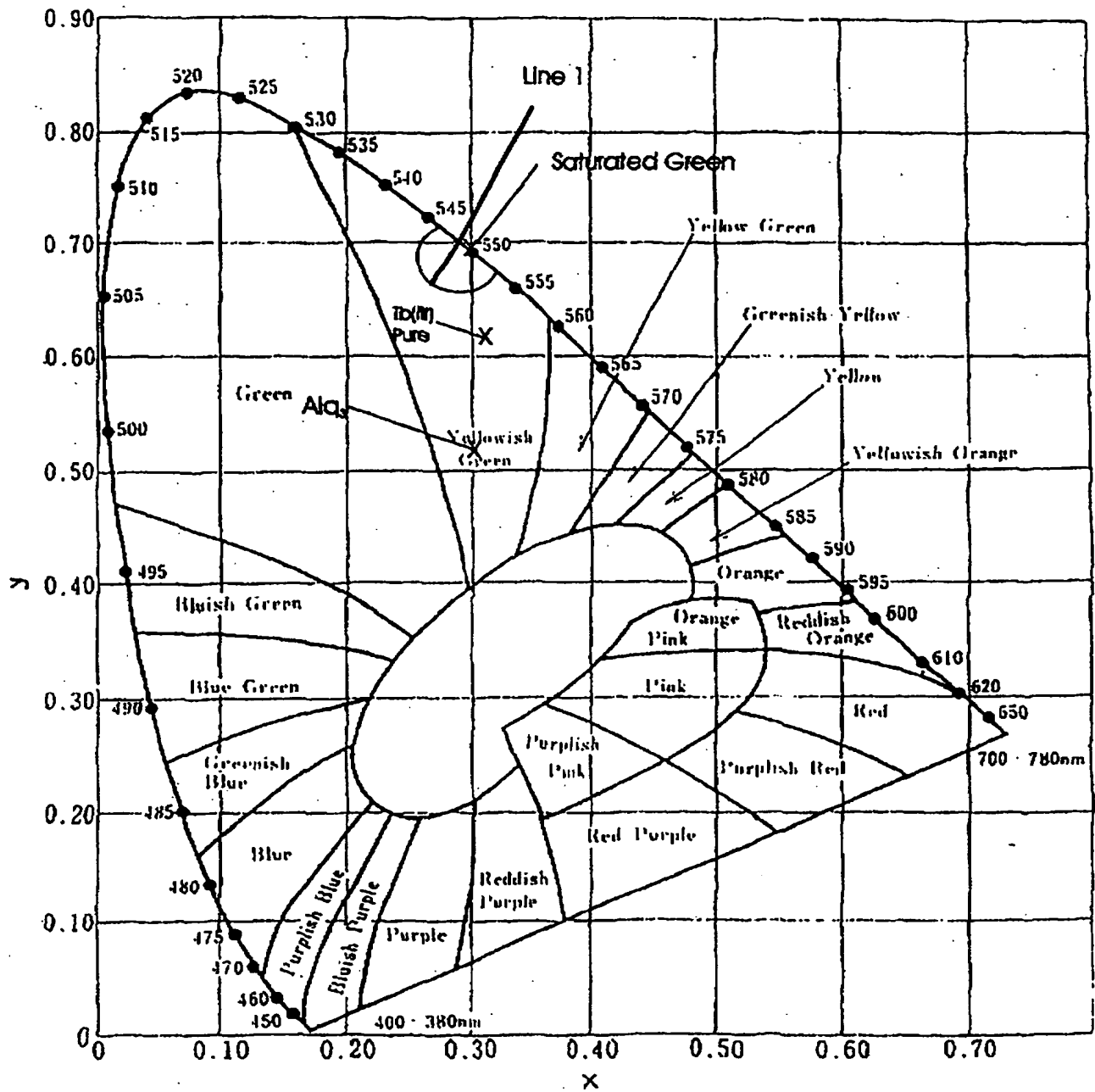


Fig. 18

INTERNATIONAL SEARCH REPORT

 Interna application No
 /GB 02/01884

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 H05B33/14 C09K11/06 C09K11/08

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 H05B C09K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ, COMPENDEX, INSPEC

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 929 104 A (SONY CORP) 14 July 1999 (1999-07-14) examples 2,6,7	1,3-8, 10-12
X	WO 00 32719 A (KATHIRGAMANATHAN POOPATHY ;SOUTH BANK UNIV ENTPR LTD (GB)) 8 June 2000 (2000-06-08) example 3 figure 3 page 3, line 14-19 page 5, line 1-15 page 7, line 28 -page 8, line 6 page 8, line 27 -page 9, line 1 --- -/--	1-8

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

5 September 2002

Date of mailing of the international search report

13.09.02

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Doslik, N

INTERNATIONAL SEARCH REPORT

Intern Application No
T/GB 02/01884

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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X	WO 00 32718 A (KATHIRGAMANATHAN POOPATHY ;SOUTH BANK UNIV ENTPR LTD (GB)) 8 June 2000 (2000-06-08) cited in the application claims 1,8,14 ---	2,4-8
A	KIDO J ET AL: "White-light-emitting organic electroluminescent device using lanthanide complexes" JAPANESE JOURNAL OF APPLIED PHYSICS, PUBLICATION OFFICE JAPANESE JOURNAL OF APPLIED PHYSICS. TOKYO, JP, vol. 3B, no. 35, pages L394-L396, XP002079654 ISSN: 0021-4922 the whole document ---	1-12
A	Y. K. KIM ET AL.: "Chlorine effect on electroluminescence of Tb complexes" SYNTHETIC METALS, vol. 111-112, - 2000 pages 113-117, XP002212372 the whole document -----	1-12

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